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# MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

## Quarterly Progress Report

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# I. SUMMARY OF PROGRESS TO DATE

## Brief Summary

### 1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff)

The book, "Construction Materials for Coal Conversion--Performance and Properties Data," has been completed, was published in September and is now available for sale from the Superintendent of Documents, Government Printing Office.

### 2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

Unit creep measurements were made on the five fused cast oxide refractories in order to identify the maximum use temperatures under static stress conditions. The materials were tested up to 1400 °C using stresses of 10 to 30 MPa (1.4 to 4.4 kpsi). Strength measurements of siliconized silicon carbide were made at temperatures up to 1400 °C preliminary to carrying out static load tests.

## II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

### 1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff)

Progress:

The book, "Construction Materials for Coal Conversion--Performance and Properties Data," has been completed. During the last quarter, the various incomplete subsections were finished and reviewed. The total manuscript was revised, reviewed by the publications division and submitted for publication. The printed books, with issue date September, 1982, are in hand and available for purchase. The book is a National Bureau of Standards Special Publication, number 642. NBS SP642 is available under stock number 003-003-02442-2 from the Superintendent of Documents, Government Printing Office, Washington, DC 20402, for $16.00 (add 25% for mailing outside the U.S.).

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

Progress:

Failure stresses and temperatures for the five fused cast oxide refractories were determined under conditions of unit creep. In this technique specimens were held under constant stress for periods of 24 hours at temperatures starting at 800 °C; and, then the temperature was raised 100 °C each 24 hours until excessive creep or failure by crack propagation occurred. Measurements were made using stresses of 10, 20 and 30 MPa for each of the materials. The results of the tests are summarized in Tables 1 and 2. The most creep resistant materials were the $\alpha$ alumina and the chrome alumina spinel, as determined from the deflection measurements.

The microstructures of two fused cast materials are shown in Figure 1. The varying grain size and porosity in the materials are clearly seen in these light micrographs. Thin sections of the materials were made for examination by electron microscopy in order to identify the phase distribution within the compacts.

Some preliminary tests were carried out on a siliconized silicon carbide material. The billet was one of five received from the Norton Company and designated NC-435. The billet was 10 x 10 x 0.6 cm and was cut into bars 4 x 5 x 50 mm for testing in four-point bending mode. Sets of 3 bars were broken at room temperature and after holding for one hour at 1200, 1300, and 1400 °C. The results are shown in Table 3. The strength decrease at 1400 °C occurred, of course, because the silicon space filling component melted. Spheres of melted silicon were found on

the fracture surface of specimens broken at 1400 °C. The silica film that formed on the exposed ground surfaces during the soaking period prevented the phase from extruding through these surfaces.

Specimens were held under a static load for 168 hours to determine any creep or strength loss as a result of stress. As seen from the tabulation in Table 4 there is a slight strength increase. There was no measurable creep during this period.

Plans:

A final report on the fused cast and silicon carbide materials will be prepared and will include the creep results as well as the microstructural analyses.

Table 1. Summary of strength obtained with dynamic load.

| Material | Modulus of Rupture, Dynamic MPa | |
|---|---|---|
| | 25 °C | 1000 °C |
| $Al_2O_3 \cdot ZrO_2 \cdot SiO_2$ Monofrax CS-3 | 93.0 ± 7.5 | 109.0 ± 13.8 |
| | 25 °C | 1100 °C |
| α + β $Al_2O_3$ Monofrax M | 38.1 ± 1.9 | 36.2 ± 3.0 |
| α $Al_2O_3$ Monofrax A | 64.6 ± 5.6 | 52.3 ± 1.8 |
| Cr-Aℓ Spinel Monofrax E | 37.0 ± 2.4 | 47.5 ± 4.3 |
| Cr-Aℓ Oxide Monofrax K-3 | 42.2 ± 3.4 | 27.3 ± 10.0 |

Table 2. Failure temperature for samples under static load.

| Material | Static Load 10 MPa Failure Temperature °C | Static Load 20 MPa Failure Temperature °C | Static Load 30 MPa Failure Temperature °C |
|---|---|---|---|
| $Al_2O_3 \cdot ZrO_2 \cdot SiO_2$ Monofrax CS-3 | 900 | 900 | 800 |
| α + β $Al_2O_3$ Monofrax M | 1400 | 1100 | * |
| α $Al_2O_3$ Monofrax A | 1300 | 1300 | * |
| Cr-Aℓ Spinel Monofrax E | 1400 | 1100 | * |
| Cr-Aℓ Oxide Monofrax K-3 | 1400 | 1100 | * |

*In progress.

Table 3. Strength of Siliconized SiC

| Temperature °C | Modulus of Rupture MPa |
|---|---|
| 25 | 367 ± 74 |
| 1200 | 439 ± 47 |
| 1300 | 376 ± 56 |
| 1400 | 91 ± 30 |

Table 4. Strength of siliconized SiC after 168 hours under static load.

| Temperature °C | Static Load MPa | Modulus of Rupture MPa |
|---|---|---|
| 1200 | 100 | 481 ± 59 |
| 1300 | 100 | 394 ± 38 |

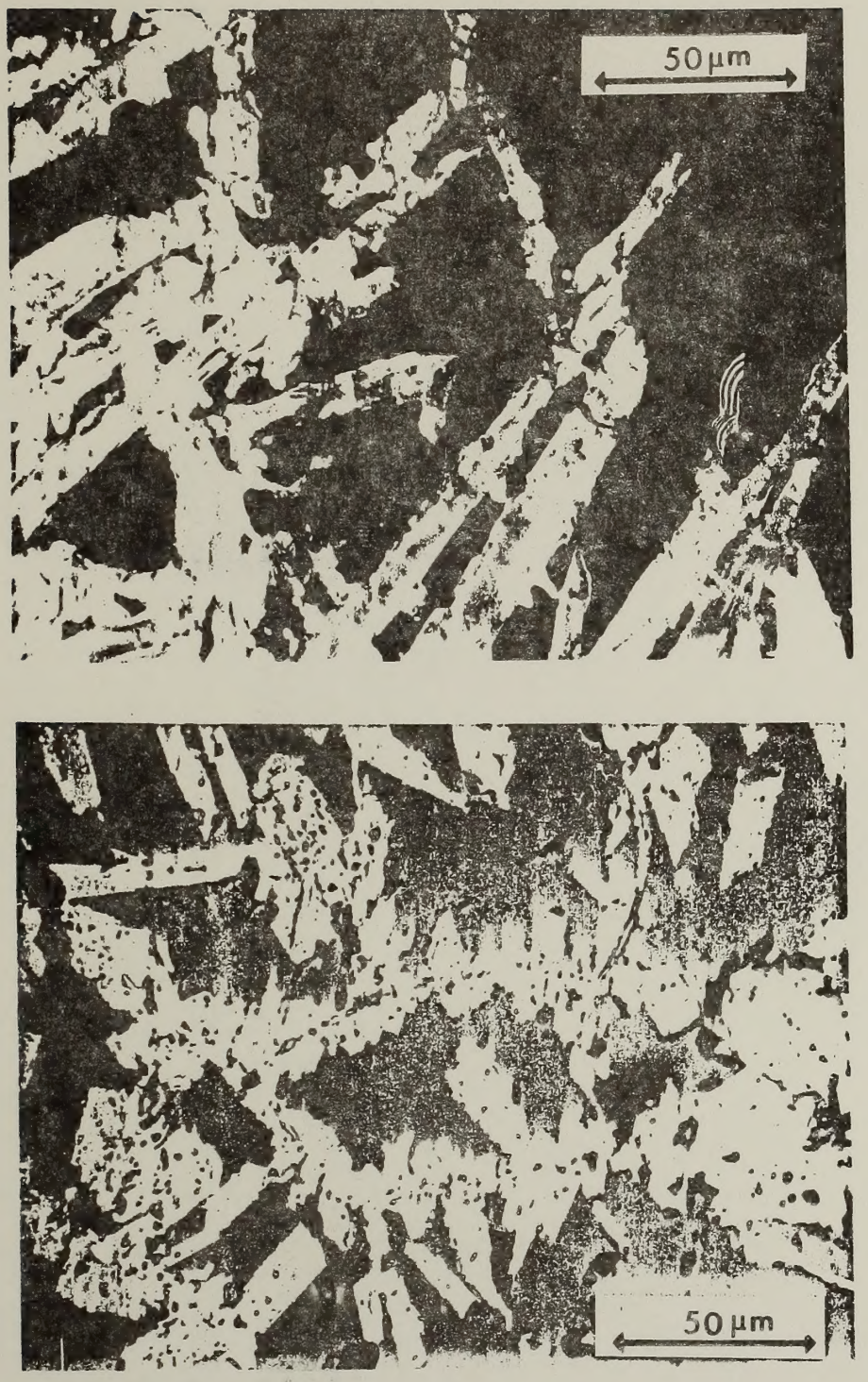


Figure 1. Light micrographs of polished sections of fused cast refractory samples. (a) alumina-zirconia-silica, showing plates of alumina and alumina containing zirconia in an alumina-silica glass; (b) chrome-alumina showing plates of chromia in a chrome alumina matrix.